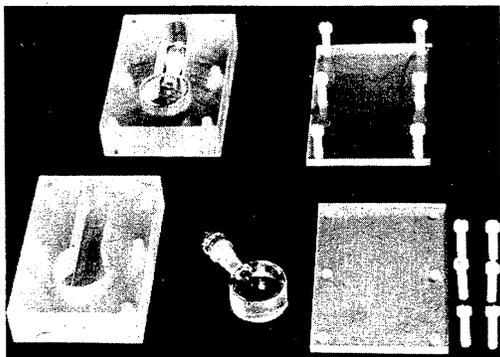


NBS Measurement Services:

Fricke Dosimetry in High-Energy Electron Beams



NBS
Special
Publication
250-4



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U.S. Department of Commerce
National Bureau of Standards

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- Dosimetry

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Engages in forefront research in nuclear and elementary particle physics; performs highly accurate measurements and theoretical analyses which probe the structure of nuclear matter; and improves the quantitative understanding of physical processes that underlie measurement science.

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PREFACE

The calibration and related measurement services of the National Bureau of Standards are intended to assist the makers and users of precision measuring instruments in achieving the highest possible levels of accuracy, quality, and productivity. NBS offers over 300 different calibration, special test, and measurement assurance services. These services allow customers to directly link their measurement systems to measurement systems and standards maintained by NBS. These services are offered to the public and private organizations alike. They are described in NBS Special Publication (SP) 250, NBS Calibration Services Users Guide.

The Users Guide is being supplemented by a number of special publications (designated as the "SP 250 Series") that provide a detailed description of the important features of specific NBS calibration services. These documents provide a description of the: (1) specifications for the service; (2) design philosophy and theory; (3) NBS measurement system; (4) NBS operational procedures; (5) assessment of measurement uncertainty including random and systematic errors and an error budget; and (6) internal quality control procedures used by NBS. These documents will present more detail than can be given in an NBS calibration report, or than is generally allowed in articles in scientific journals. In the past NBS has published such information in a variety of ways. This series will help make this type of information more readily available to the user.

This document (SP 250-4), NBS Measurement Services: Fricke Dosimetry in High-Energy Electron Beams, by C. G. Soares, E. L. Bright, and M. Ehrlich, is the fourth to be published in this new series of special publications. It describes the preparation and calibration of ferrous-sulfate (Fricke) chemical dosimeters used for the measurement of absorbed dose to water in electron beams in the energy range from 5 to 50 MeV (see test numbers 48010M and 48011M in the SP 250 Users Guide). Inquiries concerning the technical content of this document or the specifications for these services should be directed to the authors or one of the technical contacts cited in SP 250.

The Center for Radiation Research (CRR) is in the process of publishing 21 documents in this SP 250 series, covering all of the calibration services offered by CRR. A complete listing of these documents can be found inside the back cover.

NBS would welcome suggestions on how publications such as these might be made more useful. Suggestions are also welcome concerning the need for new calibration services, special tests, and measurement assurance programs.

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ABSTRACT

The NBS Fricke-Dosimetry Service (advertised in NBS Special Publication 250, 1986-1988 and earlier editions) is described in detail. After a brief historical introduction and description of the service, the theoretical basis (including what quantities are measured, how, and why) and the philosophy of internal quality checks are discussed in some detail. This is followed by a description of the physical setup and of the step-by-step operating and reporting procedures. Throughout this section, there is reference to sample records of past performance, in order to facilitate continuity of operation in the case of personnel changes. The document concludes with a discussion of the uncertainties involved in this measurement quality assurance service, safety considerations, and an appendix containing samples of all form letters and of the final report mailed to the participants.

Key words: absorbed dose to water; bi-annual service; chemical dosimetry; dose interpretation; dosimeter preparation; electron therapy; ferrous-sulfate (Fricke) solution; internal quality check; measurement quality assurance; plastic phantom; spectrophotometry

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1. DESCRIPTION OF SERVICE

1.1 History

Since 1967, the National Bureau of Standards (NBS) has been providing a measurement-assurance service for testing the status of dosimetry for electron beams in the energy range from about 5 to 50 MeV. (For details see Chapter VI, Ionizing Radiation Measurements, test numbers 48010M and 48011M — formerly 8.5B and 8.5C — of NBS Special Publication 250, The NBS Calibration Services Users Guide 1986-1988.) The service, which is directed mainly toward radiation-therapy applications in the United States, was established upon the request of the American Association of Physicists in Medicine (AAPM) at the time of the electron-therapy clinical trials, for which uniform dosimetry among all the participants was essential. Initially, it was thought that the service would be discontinued as soon as NBS could offer in-phantom calibrations for radiation-therapy instruments in terms of absorbed dose to water, or when reliable methods would be available for arriving at absorbed dose to water from exposure measurements in air [1]¹ with suitably calibrated instruments. NBS is now in a position to perform these calibrations, and reliable methods for deriving absorbed dose to water from exposure have been developed [2]. Nevertheless, the service is being continued since there is a continuing demand for the "measurement-quality assurance" it provides for the daily operation of radiation-therapy departments. During its first year, the service was subsidized by the Bureau of Radiological Health and thus could be offered quarterly and at a fraction of actual cost. Since the end of the subsidy, it has been offered semi-annually, and the charges cover the actual cost. During the past eighteen years, a total of roughly 75 groups have participated, some annually, most of them semi-annually. Most of these groups use electron beams for therapy. Occasionally we accept requests from groups desiring checks on their cobalt-60 gamma-ray dosimetry. Requests from groups desiring checks on their high-energy bremsstrahlung dosimetry are accepted only with the understanding that the NBS dose interpretation from high-energy bremsstrahlung is associated with larger uncertainties.

Over the years, descriptions of this service and reviews of results have been published. Reprints of these and other pertinent publications are filed with this document and are available to the public upon request. The following highlights are partially extracted from the publications.

1.2 The Service

Ferrous-sulfate (Fricke) dosimeters, prepared at NBS and checked by means of ⁶⁰Co gamma pre-irradiation, are shipped to the participating institutions with the request to irradiate them in a polystyrene phantom, using the dosimeter depth and the phantom and field sizes specified by the AAPM [2]. Figure 1 shows the dosimeter assembly, consisting of the ferrous-sulfate (Fricke) solution in a ground-glass stoppered, radiation-resistant, quartz spectrophotometer cell with quartz windows. The cell is cradled in a polystyrene block machined to fit into a recess of the participant's polystyrene

¹Numbers in brackets indicate the literature references at the end of this paper.

phantom. The dosimeters are shipped pre-paid by a commercial delivery service. Since they contain a corrosive liquid in unsealed containers they cannot be sent by mail, and, if air transport at high altitudes is involved, the packages must be marked for transportation only in the pressurized compartment of the plane. (See also Section 4.)

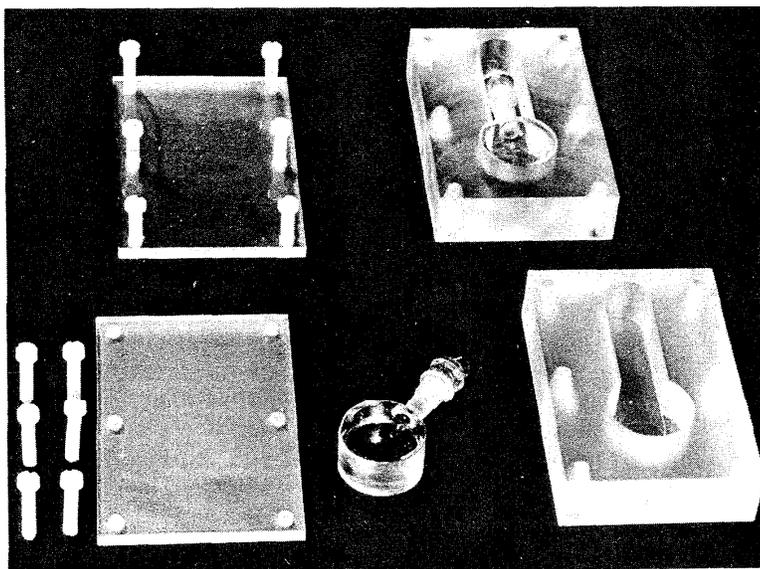


Figure 1. Dosimeter Assembly [3]. The stoppered spectrophotometer cells are cradled in a 5.1 cm x 7.6 cm x 2.0 cm (high) polystyrene block, with a styrofoam plug (not shown) wedged in above the stopper. The 3-mm polystyrene lid is presently screwed on with two rather than with the six screws shown.

The participants irradiate all but one of the furnished dosimeters to between 50 and 80 Gy (5000 and 8000 rad) to water, usually at electron energies between 5 and 50 MeV, employing the specified irradiation geometry.

After irradiation, the participants return the dosimeters to NBS for spectrophotometric evaluation of the ferric-ion concentration in terms of absorbed dose to water, using a value for the radiation-chemical yield compatible with the most recent ICRU recommendations [4]. Return shipment is prepaid by participants, and the restrictions on delivery service and mode of transportation are the same as for shipment from NBS. The participants are asked to return the dosimeters within three working days from their receipt, if at all possible. Once the evaluation is completed, NBS informs each participant by mail of his own test results and of the results of all other participants, but without divulging their identities. In addition, regular participants actively engaged in radiation therapy are alerted by telephone if their dose interpretations are badly out of line, say, by 20 percent or more. All participants are free to discuss their results and possible problems with NBS, either by mail or by telephone.

Appendix 1 contains all forms used in written exchanges with participants. A sample of a final report is shown in Appendix 2.

2. THEORY AND DESIGN PHILOSOPHY

2.1 Theory

The ferrous-sulfate (Fricke) dosimetry system has been discussed extensively in the literature.[5] The chemical changes it undergoes upon exposure to ionizing radiation are well understood and lead to changes in optical properties that are readily and accurately measurable by relatively simple optical means. Because its net optical absorbance is proportional to the absorbed dose to the system over a wide range of irradiation levels, and the system consists mainly of water, it lends itself well to a determination of absorbed dose to water, which is the quantity of interest in radiation therapy.

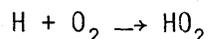
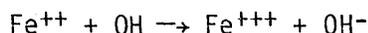
NBS uses the following conventional dosimeter ingredients²:

0.001 M $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, dissolved in

0.8 N H_2SO_4 , well aerated, and

0.001 N NaCl.

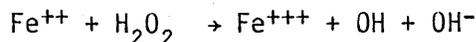
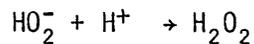
The NaCl desensitizes the system against organic impurities but causes an increase in the rate dependence of its radiation-chemical yield. Therefore, participants using pulsed electron beams are asked to irradiate at dose rates not exceeding 10 Gy per microsecond pulse or a total of 350 Gy for any train of microsecond pulses [5]. A simplified reaction mechanism (i.e., one that does not consider the modifications caused by NaCl) is shown below; it demonstrates how aeration (i.e., the production and maintenance of a sufficient amount of O_2 at all times) enters into the reaction and determines the Fe^{+++} yield:



²Meaning of the symbols "M" and "N":

The symbol M: The mole is the molecular weight of a substance expressed in grams. The molarity of a solute is the number of moles of the solute contained in 1000 cm^3 of solution. Therefore, a one-molar (1 M) solution of sulfuric acid contains 98.08 g of sulfuric acid per 1000 cm^3 of solution.

The symbol N: The normality of a solution is its equivalent gram-molecular weight concentration. The equivalent gram-molecular weight of an acid is its molecular weight expressed in grams, divided by the number of hydrogen atoms per molecule. Therefore, a normal (1 N) solution of sulfuric acid contains 49.04 g of sulfuric acid per 1000 cm^3 of solution.



Ferric-ion yield is defined as the number of Fe^{+++} ions formed in the Fricke solution per unit of absorbed dose to the solution. It may be determined spectrophotometrically with high accuracy by the change in absorbance of the solution, which is the logarithm to the base ten of the ratio of incident to transmitted radiant energy Flux. Absorbance is proportional to the molar concentration of the Fe^{+++} ions and the optical pathlength, with the proportionality constant referred to as the molar linear absorption coefficient, or the molar extinction coefficient (Beer's law). The change in optical density measured with a spectrophotometer with good optical properties is equal to the change in absorbance. In general, absorbance depends on the wavelength and the temperature at which the measurements are made. At a given temperature and wavelength, the average absorbed dose to the Fricke solution, \bar{D}_F , then is obtained from the change in absorbance, ΔA , as:

$$\bar{D}_F = \frac{\Delta A}{\rho \ell \epsilon G(\text{Fe}^{+++})} \quad (1)$$

where ΔA is the change in absorbance, ρ is the density of the Fricke solution, ℓ is the optical pathlength, ϵ is the molar linear absorption coefficient, and $G(\text{Fe}^{+++})$ is the Fe^{+++} ion yield.

The average absorbed dose to water, \bar{D}_W , is obtained as [4]

$$\bar{D}_W = 1.004 \bar{D}_F \quad (2)$$

For measurements of absorbances at 25°C, at the 304-nm absorption peak, at an optical pathlength of 0.01 m, and assuming a Fricke-solution density of 1024 kg m⁻³ and a product of $\epsilon G(\text{Fe}^{+++}) = 352 \times 10^{-6} \text{ m}^2 \text{ kg}^{-1} \text{ Gy}^{-1}$ [4], the expressions reduce to

$$\bar{D}_F = \frac{\Delta A}{1024 \times 0.01 \times 352 \times 10^{-6}} \text{ Gy} = 277 \Delta A \text{ Gy} \quad (3)$$

and

$$\bar{D}_W = \bar{D}_F \times 1.004 = 278 \Delta A \text{ Gy} \quad (4)$$

2.2 Design

At the time the service was first implemented as a stop-gap procedure, simplicity of design and operation was of the essence. Making the expensive but re-usable stoppered quartz spectrophotometer cells an integral part of the Fricke dosimeters (see fig. 1) reduces handling time and possible sources of contamination of the Fricke solution.

An alternative would be to transfer the solution first into specially cleaned, disposable borosilicate ampoules; flame-seal the ampoules without damage to the solution from contact with gases escaping from the heated glass; and keep the solution in the sealed ampoules until ready for final readout. The solution would then be transferred once more, this time into a spectrophotometer cell that would remain in the spectrophotometer and would be filled, rinsed and re-filled until the contents of all the ampoules was read out. Such a procedure seemed too elaborate for a stop-gap service, but the adopted system incorporating unsealed dosimeters also has its draw-backs. Because the Fricke solution is corrosive, the unsealed dosimeters have to be shipped by carriers other than the U.S. Mail and flown either at low altitude or in pressurized compartments. Fricke solution in unsealed cells also is more prone to succumb to influences of the environment than solution in sealed ampoules, causing a larger dependence of spurious absorbance growth³ on dosimeter history. The resulting additional uncertainty in the dose interpretation would be unacceptable in a NBS calibration service, but is acceptable in a service with the primary function of having the participants compare their ability to measure absorbed dose to water with that of their peers.

The service was designed to be run on a small scale (offered twice a year, not over forty participants at a time), with manual dosimeter preparation and automation only in the spectrophotometric-readout and data-handling stages. A discussion of a possible upgrading and expansion of the service is given in section 2.4.

2.3 System Operation

2.3.1 Statistical distribution of absorbance growth in the NBS unsealed system.

Prior to the start of the service, an experiment was performed to ascertain the influence of irradiation level and storage conditions on absorbance growth during a period of about four weeks between preparation and final readout of the Fricke solution kept in the stoppered spectrophotometer cells. The cells were irradiated at NBS to different exposure levels, and one-half of them were then shipped cross-country while the other half remained at NBS. There was no significant dependence of spurious absorbance growth on exposure level (and thus on absorbance level), and there was no significant difference between the average absorbance growth of shipped cells and those remaining at NBS, the average absorbance growth amounting to between 0.003 and 0.004, which corresponds to between 0.8 and 1.1 Gy to the solution. Over the years, a record has been kept of the standard deviation of the average absorbance growth on identically pre-irradiated Fricke-dosimetry control cells, some of which were shipped and the rest remained at NBS. The distribution of the absorbance growths has a tendency to be more symmetrical for the shipped batch than for the batch remaining at NBS, which shows a "tail" on the side of larger absorbance growth.[3]

³In this paper, the term "spurious absorbance growth" is used to mean absorbance growth not caused by ionizing radiation but, e.g., by auto-oxidation.

2.3.2 Internal quality check on performance of individual dosimeters.

In order to check on the performance of the Fricke dosimeters prior to their use, it was decided to pre-irradiate all the freshly-filled spectrophotometric cells, about 50 or 60 at a time, with ^{60}Co gamma radiation to a level of between 40 and 50 Gy to water. This procedure was adopted after it had been established [3] that the response of the dosimeters to ^{60}Co gamma radiation and to high-energy electrons was additive. Irradiation and readout of each dosimeter before actual use permits (a) a statistical study of the random variations from dosimeter to dosimeter in the response to a given irradiation level, as measured spectrophotometrically; (b) the detection and elimination from further use of dosimeters with responses far removed from the mean for the particular batch; and (c) the evaluation of the absorbed doses administered by the participants from higher (and therefore more precise) absorbance levels. If a system employing sealed ampoules were adopted in the future, the advantages of individual dosimeter readouts prior to shipment would be lost since the ampoules would have to be re-opened and the solution transferred to spectrophotometer cells for readout. Pre-irradiation of all dosimeters could continue if raising the absorbance level for the readout were still found to be of advantage, but a portion of each solution batch would have to be set aside for pre-irradiation as an internal quality check.

Since the dosimeters can be used for determining doses up to more than 300 Gy while participants are being asked to deliver only about 50 Gy, it is in principle possible to use the same set of dosimeters up to six times, as long as linearity between optical absorbance and absorbed dose has been established to hold for the particular batch over the range of absorbances employed. In practice, the dosimeters so far have never been used for more than one test series. But, since 1983, the number of dosimeters requested for any one series has exceeded the number of dosimeters available. Therefore, two consecutive mailings, about six to eight weeks apart, were made per series, using some of the dosimeters for a second round of irradiations, with satisfactory results. However, there are some indications that the reproducibility of the dose interpretation decreases slightly with consecutive dosimeter use because of an increase in spurious absorbance growth and growth variability. For this reason, the number of available spectrophotometer cells and accessories was increased during 1986.

2.3.3 Internal quality checks on spectrophotometry [6]

Wavelength scale. Because of spectrophotometer variability it is recommended that the spectrophotometer wavelength scale be periodically checked. It was decided to perform this check prior to each series of spectrophotometer readouts by determining the wavelength setting near 304 nm for which the ferric-ion absorbance shows a maximum, and to make all readouts at this wavelength.

Molar linear absorption coefficient for ferric ions. It is further recommended that the molar linear absorption coefficient for ferric ions for the particular spectrophotometer in use be determined. This procedure has been followed throughout the years the Fricke-dosimetry service has been in operation, re-determinations having been performed whenever there was a change in spectrophotometer, spectrophotometer components or spectrophotometer adjustment. The preparation of the ferric-ion solution is described in detail in Appendix S2 to the ASTM Standard [6].

ICRU recently recommended the use of a fixed product of the molar linear absorption coefficient and the ferric-ion yield.[5] The value recommended for the product agrees to within 1 percent with the value based on our measured molar linear absorption coefficient and the ferric-ion yield used by us through 1985 [7]. The recent ICRU recommendations have now been incorporated in our procedure and are reflected in the constants given in eqs. (3) and (4) (section 2.1). However, we plan to continue the practice of re-determining the molar linear absorption coefficient experimentally when the need arises.

Constancy and Linearity. As a check on the constancy and linearity of the spectrophotometer over the one- to two-month period of any given test series, it was suggested that an independent check be kept on these parameters by means of absorbance measurements on several dilutions of a "1 g%" solution of potassium nitrate (i.e., an aqueous solution containing 1 g of potassium nitrate per 100 cm³), which has an absorption peak at a wavelength of about 302 nm, i.e., close to the ferric-ion absorption peak used for Fricke dosimetry.[8] Since then, Standard Reference Material SRM 2031, consisting of metal-on-quartz filters for the verification of spectrophotometric absorbance scales in the ultraviolet, has become available.[9] This material may replace the potassium-nitrate solution in the future.

2.4 Upgrading the Service

In preparation for a possible upgrading of the Fricke-dosimetry measurement assurance service to a calibration service, switching to a system employing sealed dosimeters is contemplated if additional experiments prove that such a system would lead to a more uniform spurious absorbance growth and therefore to a higher reproducibility in dose interpretation from absorbance differences. For this purpose, a semi-automatic ampoule-sealing unit was constructed. Its design is patterned after a unit in use by the National Physical Laboratory in Teddington, England. Also, thought is being given to automating the rinsing and filling operations of the spectrophotometer cells or glass ampoules.

Further preparations for upgrading the service would entail studies for obtaining the corrections required to produce adequate agreement with the results of calibrations in terms of absorbed dose to water by other methods. Such corrections may be needed because of the physical inhomogeneities introduced in the dosimeter-phantom system by the presence of glass and of an air space. Upgrading the service may also require at least a temporary increase in the staff assigned to the project during the preparation period, and probably some permanent staff reassignments or additional chemistry training of the present staff.

3. DESCRIPTION OF SYSTEM AND OPERATING PROCEDURE

3.1 The Physical Setup. (Most of the items enumerated are shown in the figures of sections 3.2 and 3.3.)

At the time this document is being prepared, the setup consists of:

- around 150 cylindrical 10-mm pathlength quartz spectrophotometer cells with quartz windows and ground-glass stoppers;
- Lucite racks, built to order in the NBS Shops, each holding 10 spectrophotometer cells in two rows of five, each row being identified by a letter and each cell location by a number;
- the chemicals: concentrated (96%) sulfuric acid, sodium chloride, ferrous-ammonium sulfate, and potassium nitrate, all of reagent grade;
- two glass-stoppered 2000-cm³ glass volumetric flasks for preparing and storing the Fricke solution;
- one glass-stoppered 500-cm³ glass volumetric flask for preparing and storing the potassium nitrate solution;
- two 500-cm³ glass wash bottles, each with plastic tubing and a rubber balloon for pressure filling the cells with either specially purified distilled water⁴ or Fricke solution through a glass tube drawn into a narrow nozzle;
- a suction pump with plastic tubing connected to a small glass bottle with dropping pipette for emptying the cells;
- access to a water-purifying setup;
- access to two electronic scales, one having a 0.1 kg capacity and 1-mg scale divisions, and the other having an ~ 1-kg capacity;
- an acrylic platform with a surface ruled in 2.5-cm squares for supporting the spectrophotometer cells horizontally in a vertical ⁶⁰Co gamma-ray beam;
- access to an ultrasonic cleaner equipped with a stainless-steel liner;
- access to an up-to-date, computer-compatible spectrophotometer with good optical properties, equipped with temperature-control for the cell compartment;
- access to a micro-computer, for data acquisition and data handling; and
- various accessories, e.g., three dust-free cabinets, a graduated glass cylinder, beakers, a weighing dish, a spatula, tweezers with flexible tips, a funnel.

⁴Unless otherwise noted, the distilled water referred to later always is the specially purified distilled water produced in the water-purifying setup, that was used to prepare all the solutions.

Initially, all glassware to be used for the Fricke solution was cleaned with hot concentrated sulfuric acid, the bottles were filled with the acid and then heated on a hot-plate until the acid almost boiled; the other items were immersed in almost boiling sulfuric acid and rinsed several times in distilled water before and after the sulfuric-acid cleaning procedure. The spectrophotometer cells and their stoppers were initially rinsed several times with distilled water. After being placed in their racks, they were filled several times with distilled water and emptied between fillings alternately by suction and shaking. The cells then were filled with 0.8-normal sulfuric acid and stoppered; racks and cells were immersed up to the cell necks in a bath of singly-distilled water for 15 minutes of ultrasonic cleaning.

Also, the cross section of the ^{60}Co gamma-ray beam in room B034 used for the pre-irradiation of all Fricke dosimeters was initially mapped at the irradiation distance of 147.9 cm from the source by means of readings of a laboratory-standard ionization chamber placed on the support platform and moved across the beam cross section in 2.5-cm steps to obtain a grid of absorbance-correction factors for all Fricke-dosimeter locations. The resulting array of correction factors is shown in figure 2 for the source and distance used through 1985. At the time of preparation of this document, the source is being replaced by a source of higher activity in room B035, in order to reduce the irradiation time.

The procedures discussed above are not repeated prior to each subsequent use. The stoppered cells (still filled with Fricke solution, and sitting in their racks), the wash bottles, the beaker for washing the stoppers, and utensils such as weighing dish, spatula, tweezers, suction bottle, are stored in the dust-free cabinets whose doors had been replaced by cheese cloth to prevent condensation of water on the stored articles. Prior to the preparation of a new set of dosimeters, all glassware is thoroughly rinsed with distilled water.

3.2 Preparation of Dosimeters and Check on their Performance

Preparing the Fricke solution, filling the spectrophotometer cells and starting the performance check (pre-irradiation of the first batch of between 50 and 60 cells) takes at least one day. Prior to starting, at least six one-gallon well-cleaned brown glass bottles equipped with screw caps lined with aluminum foil are filled with the special distilled water from the water-purification system maintained by the Dosimetry Group of the Ionizing Radiation Division. The system presently consists of one high-capacity Raven still and the Milli-Ro 60 system of the Millipore Corporation, equipped with one organic absorption stage, two deionizing stages and one filtration stage. Instead of giving a lengthy description of the procedure, copies are shown in the following of typical pages from data books, giving the step-by-step record of the procedures for preparing the Fricke dosimeters and carrying out the internal quality checks on dosimeter performance.

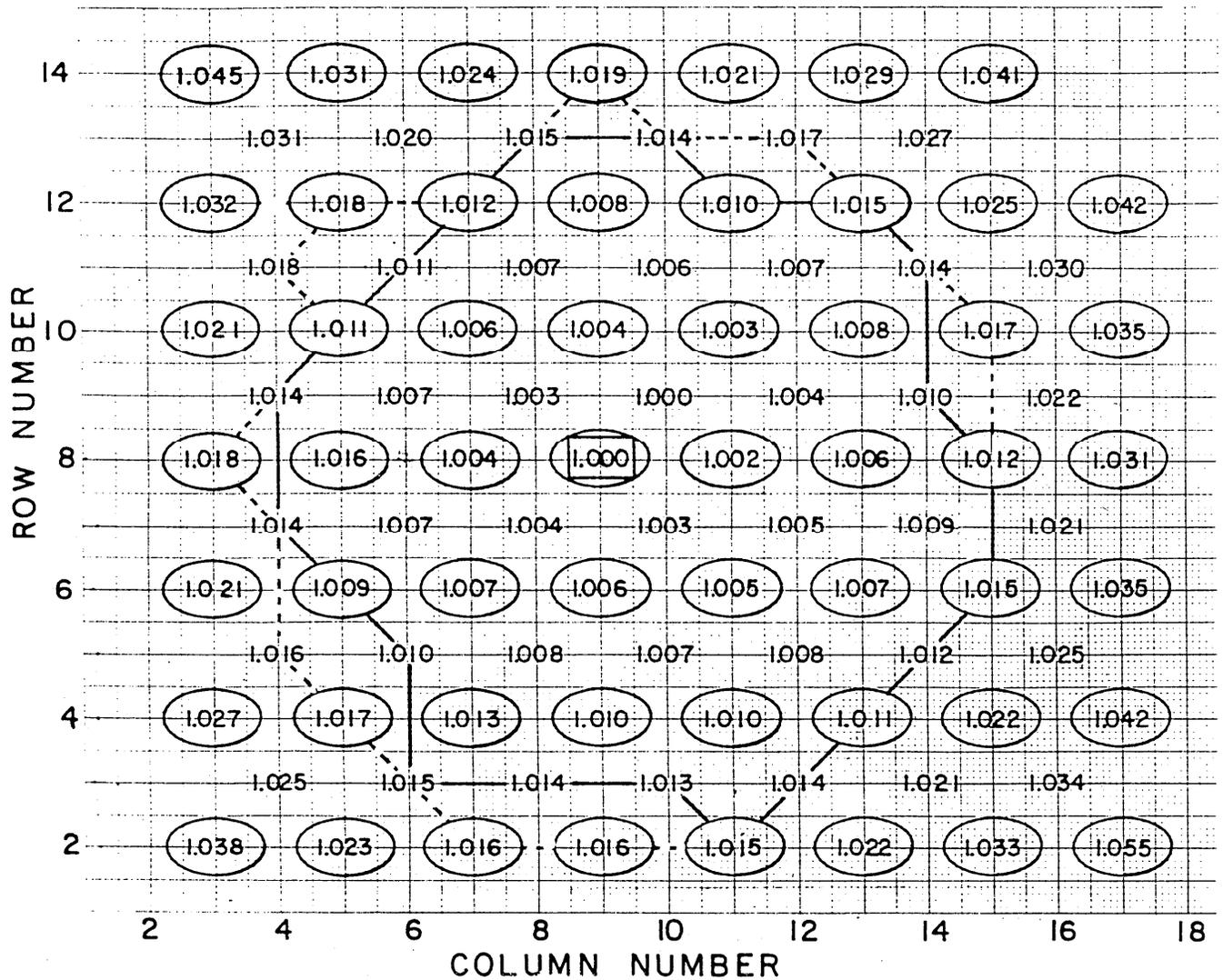


Figure 2. Mapping the Cross Section of the Cobalt-60 Gamma-Ray Beam in Room B034, Used for Pre-Irradiation; scale: 1 cm = 2.5 cm

- encircled numbers: correction factors from data points
- other numbers: interpolated correction factors
- solid-line envelope: 52 locations with corr. factors < 1.015
- dashed line: extending envelope to include 10 further locations, with corr. factors < 1.019

3.2.1 Preparing the solutions

(a) Weighing the ingredients (sample from Data Book 837, pp. 160 and 163)

Sulfuric Acid, Baker reagent grade, 96.3 percent, fresh (see fig. 3) ⁵

graduate cylinder in beaker, tared on Mettler balance model H 1200

poured ~ 46 cm³ of H₂SO₄ into cylinder

weight of acid 84.45 g

delivered 84.05 g into 2000-cm³ flask A ⁶

graduate cylinder in beaker, tared again

poured ~ 46 cm³ of H₂SO₄ into cylinder

weight of acid 83.80 g

delivered 83.80 g, into 2000-cm³ flask B ⁶

Ferrous-ammonium sulfate, same bottle as for last test series, ⁵ weighed
on Mettler balance, model H 16 (see fig. 4)

dish alone 4.13160 g

the salt 0.78430 g

should be 4.91590 g

is 4.91585 g, into 2000-cm³ flask B ⁵

dish alone 4.13162 g

the salt 0.78430 g

should be 4.91592 g

is 4.91596 g into 2000-cm³ flask A ⁵

Sodium chloride, same bottle as for last test series, ⁵ weighed on Mettler
balance H 16 (see fig. 4)

dish alone 4.13162 g

the salt 0.11690 g

should be 4.24852 g

is 4.24855 g, into 2000-cm³ flask B ⁵

dish alone 4.13163 g

the salt 0.11690 g

should be 4.24853 g

is 4.24894 g, into 2000-cm³ flask A ⁵

dish alone 4.13156 g

⁵All reagents are periodically replaced.

⁶Prior to adding the weighed ingredients, all flasks had been filled partially with water. Note that, throughout the text, the term "water" unless otherwise stated is used to mean the specially distilled water from the water purification system described in section 3.2 (p. 14).

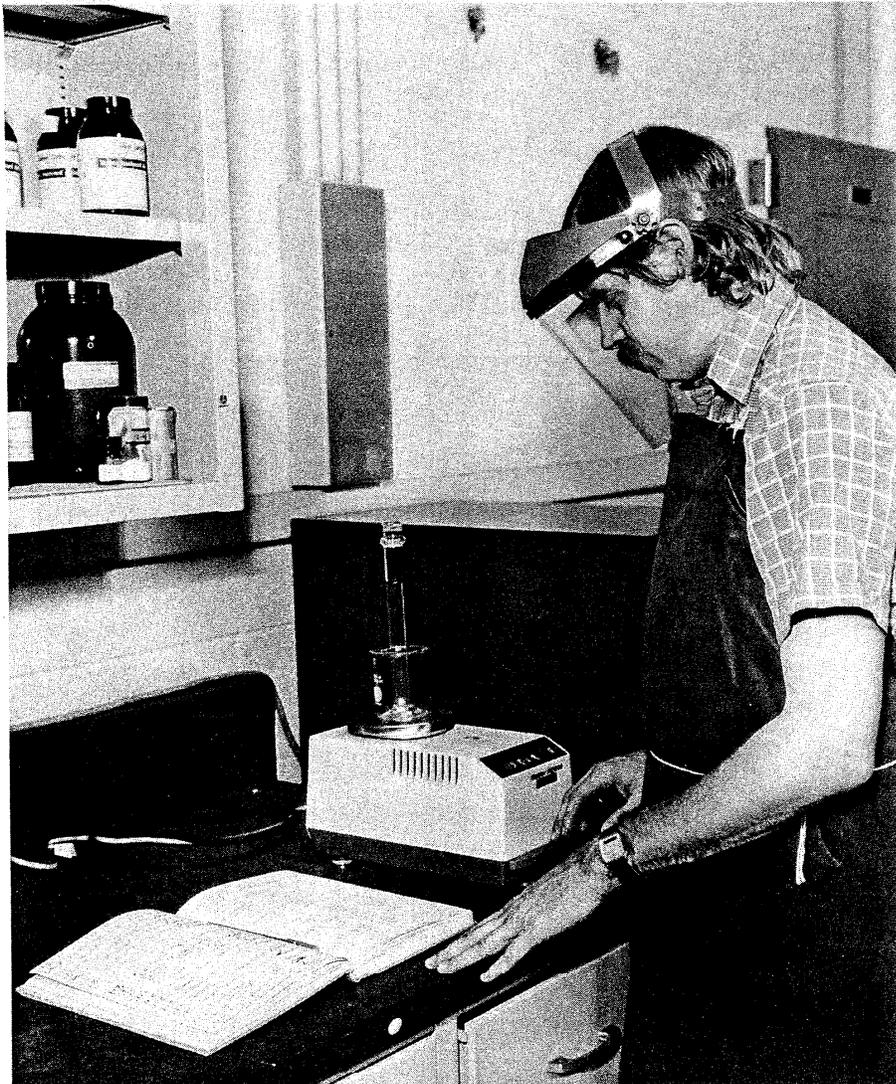


Figure 3. Weighing the Sulfuric Acid
Note rubber apron and face shield.

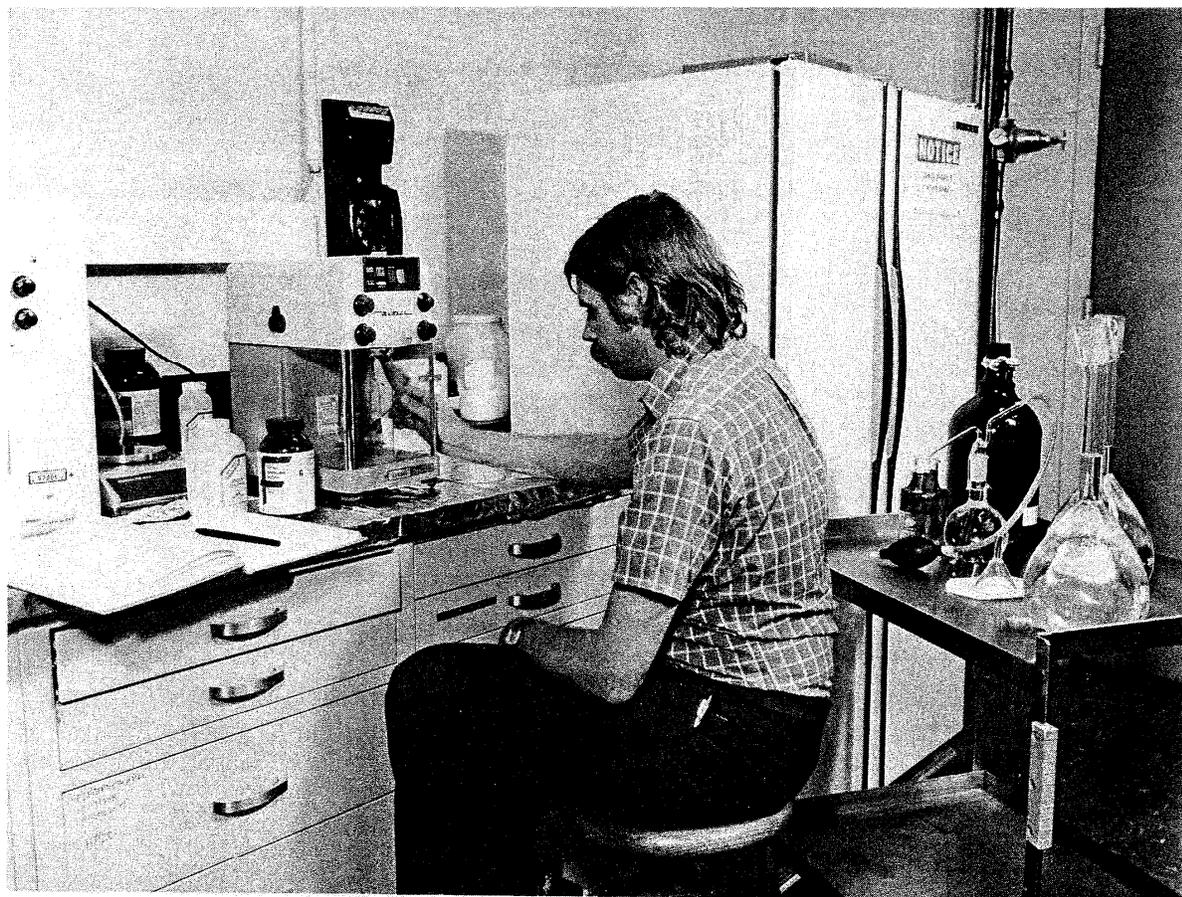


Figure 4. Weighing the Salts.

The weighed portions are transferred quantitatively from the weighing dish into the volumetric flasks through a clear, dry funnel, and washed down with water from the wash bottle.

Potassium nitrate, same bottle as for last test series, weighed on
Mettler balance H 16⁷

dish alone	4.13376 g	⁸
the salt	5.00000 g	
should be	9.13376 g	
is	9.13366 g	into 500-cm ³ flask ⁹
dish alone	4.13350 g	

All flasks filled to neck junction with water at about 11:00 a.m.

(b) Finishing the stock solutions

After having allowed enough time for all solid ingredients to dissolve completely and for the solution to return to room temperature:

- All volumetric flasks are filled with water to their marks and stoppered;
- The Fricke-solution flasks are shaken about 50 times, by turning them upside down and back again with a rotary motion (see fig. 5); during this procedure, stoppers are loosened periodically to permit air pressure to equalize.

3.2.2 Filling the spectrophotometer cells (see fig. 6)

Sample of Cell-Rinsing and -Filling Record (from Data Book 837, p. 161)

(a) Rinsing with Water

cells sucked out and filled with water, finished 11 a.m.; 10 min. rest
cells shaken out and filled with water, finished 11:20 a.m.; 10 min. rest
cells sucked out and filled with water, finished 1:30 p.m.; 10 min. rest
cells shaken out and filled with water, finished 1:46 p.m.; 10 min. rest.

⁷In 1986, the function of this solution was taken over by a standard reference material. (See section 3.3)

⁸A close to 1.00000 g% solution was required at the time, because the solution was used for a long-term constancy check of the spectrophotometer's absorbance scale. To prepare such a solution, both the weighing dish and the salt have to be in temperature and relative-humidity equilibrium with the balance (and the room). Repeated weighings, about one minute apart, have to be made until weight constancy is reached. This procedure is not required when the potassium nitrate solution is used simply to establish the location of the absorbance peak and of absorbance-scale linearity.

⁹Prior to adding the weighed ingredients, the flask was filled partially with water.

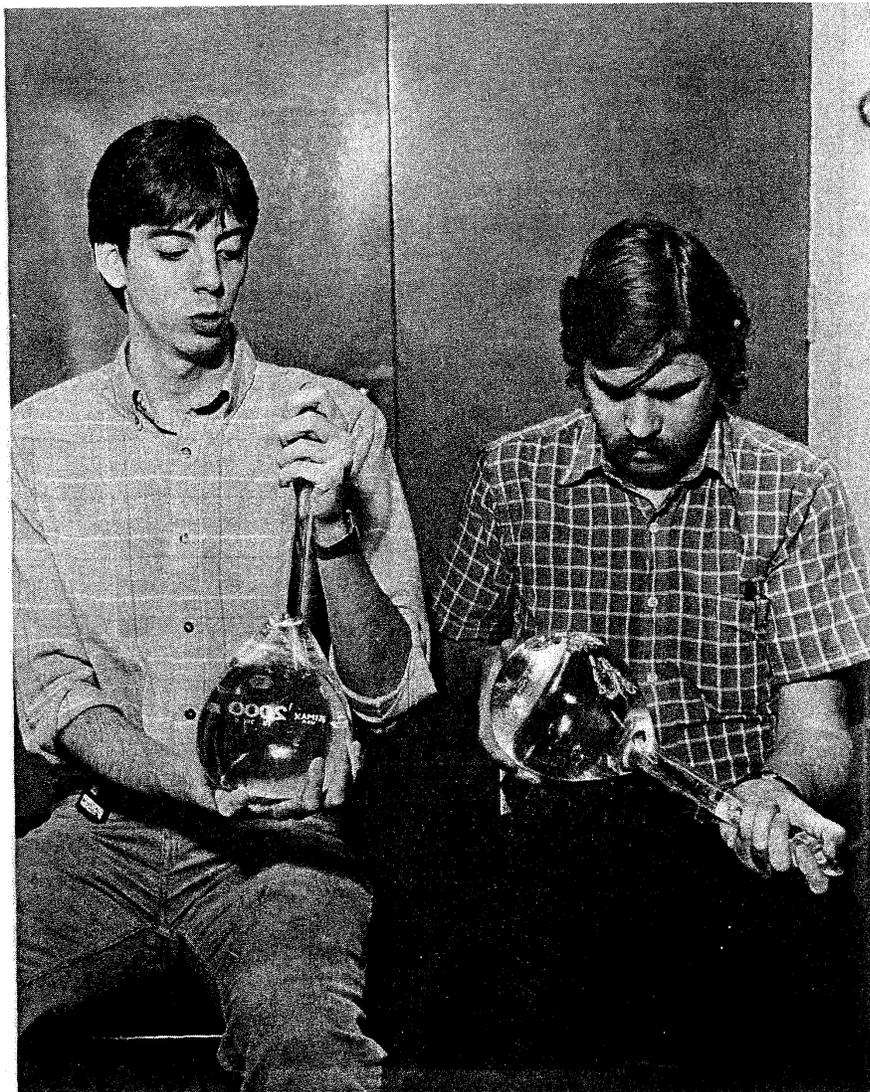


Figure 5. Aerating the Solution.

Aerating prior to filling of individual spectrophotometer cells ensures the same degree of initial aeration of the dosimeters. Normally, rubber aprons are worn during this process.



Figure 6. Filling the Spectrophotometer Cells with Fricke Solution.
Singly distilled water bathes the racks and the outside of the cells.

(b) Ferrous-Sulfate Rinses

cells sucked out and filled, finished 2:08 p.m.; 10 min. rest
cells sucked out and filled, finished 2:32 p.m.; 10 min. rest
cells sucked out and filled, finished 3:00 p.m.; 10 min. rest
last (final) fill, completed at about 3:40 p.m.

All cells stoppered by about 4 p.m.; racks and cells washed with singly distilled water; let dry overnight.¹⁰

3.2.3 Pre-irradiation with cobalt-60 gamma rays

Prior to pre-irradiation, spectrophotometry is done on the cells of one rack. (Spectrophotometry is discussed in more detail in section 3.3.) This furnishes the average background absorbance required for obtaining the net absorbance of the pre-irradiated cells for a check on the G-value of the particular batch of Fricke solution.

Figure 7 shows the pre-irradiation setup in room B 034, used through 1985; figure 2 (page 10) the associated beam cross section; and table 1 a sample of the absorbance record in one rack, obtained prior to irradiation.

Table 1. Sample, Absorbances of Cells Before Pre-irradiation, Required for Check on G-Value and ϵ G Product, from Data Book 837, page 99.^a

Cell Designation	Absorbance (Average of 15 readings, and standard deviation)	
	Side a	Side b
U-1	0.0417 ± 0.0001	0.0419 ± 0.0001
U-2	0.0415 ± 0.0001	0.0413 ± 0.0001
U-3	0.0416 ± 0.0001	0.0418 ± 0.0001
U-4	0.0389 ± 0.0001	0.0399 ± 0.0001
U-5	0.0416 ± 0.0001	0.0431 ± 0.0001
V-1	0.0397 ± 0.0001	0.0379 ± 0.0001
V-2	0.0400 ± 0.0001	0.0414 ± 0.0001
V-3	0.0424 ± 0.0001	0.0431 ± 0.0001
V-4	0.0397 ± 0.0001	0.0404 ± 0.0001
V-5	0.0444 ± 0.0001	0.0435 ± 0.0000

^aAll readings done with a slit width corresponding to 2 nm in wavelength, at a wavelength setting of 301 nm and a temperature of 25.00 ± 0.05°C. The designations, "side a" and "side b," identify the cell windows facing the ultraviolet source. We routinely reverse the cells for the second reading since specks of dirt on the cell windows may influence the reading to a different extent in the two geometries.

¹⁰If desired, irradiation of first batch of 50 to 60 cells may be started immediately after drying the cells individually with a lint-free cloth.

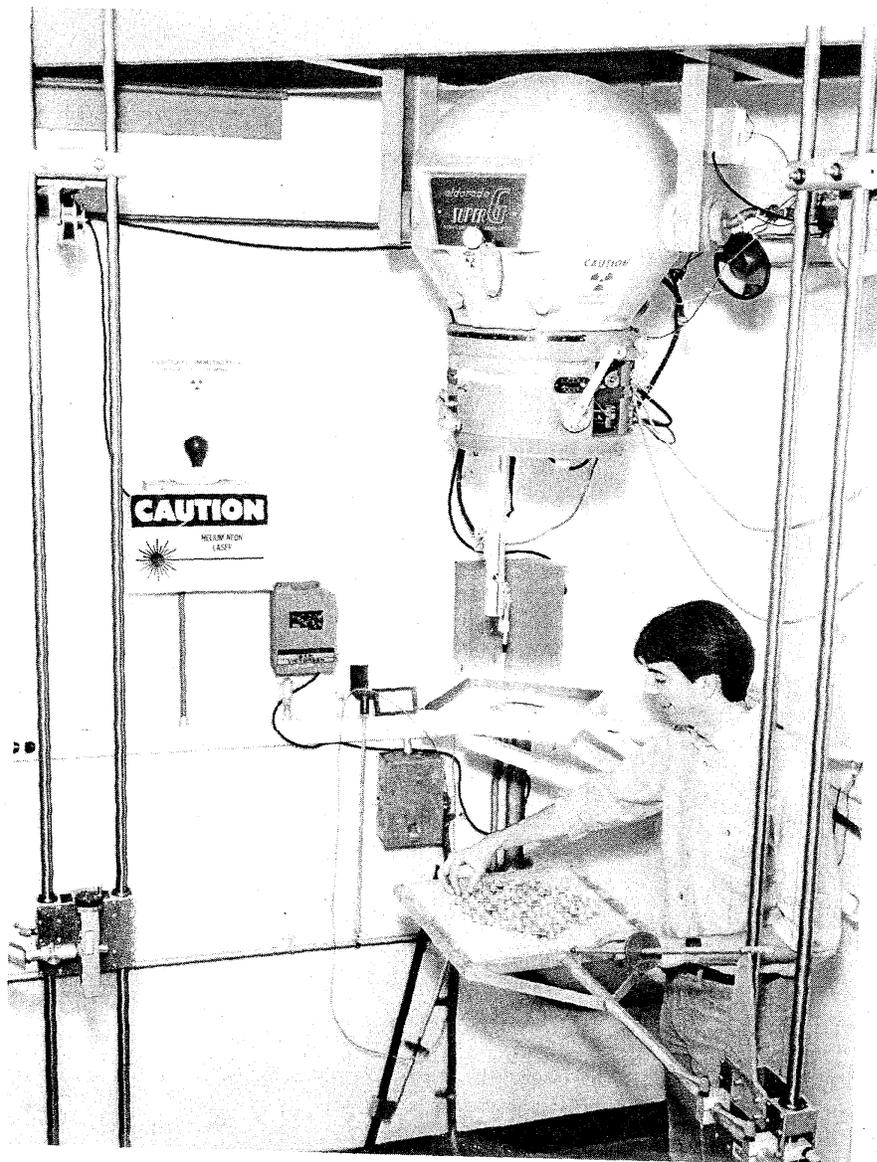


Figure 7. Pre-irradiation with Cobalt-60 Gamma Radiation in Room B034.

The surface of the support is ruled, with rows and columns marked as in figure 2 (page 15). The fixed-distance support visible at shoulder height is not in the beam.

Sample of Pre-irradiation Record (from Data Book 856, p. 39)

Exposure rate on day of last calibration (April 24, 1985): 46.98 R/h.

Exposure rates in air, at point of cell irradiations:

On November 6, 1985, $\dot{\lambda} = 46.98 e^{-0.0003601 \times 195} = 43.79$ R/h.

On November 10, 1985, $\dot{\lambda} = 46.98 e^{-0.0003601 \times 199} = 43.73$ R/h.

The number 0.0003601 is the decay constant for the cobalt-60 gamma ray source, and 195 and 199, respectively, is the number of days from the last calibration to roughly the middle of the planned irradiations.

Corresponding absorbed-dose rates to the Fricke solution:

On November 6, 1985, $\dot{D}_F = 43.79 \times 0.961 = 42.09$ rad/h (0.4209 Gy/h).

On November 10, 1985, $\dot{D}_F = 43.73 \times 0.961 = 42.03$ rad/h (0.4203 Gy/h).

The factor, 0.961, was obtained empirically as the ratio of D_F/X ,¹¹ averaged over sixteen determinations during the period of 1967 through 1974. (See Data Book 767, page 34.) D_F was computed from measured net absorbance, using the recommended G-value for cobalt-60 gamma rays and the measured value of the molar linear absorption coefficient (see table 5, page 23).

For each of the two sets of irradiations, a record is kept of the following data: Time set and time delivered; date and time when timer was started and date when timer stopped; timer reading at beginning and end of irradiation; total dose delivered to the Fricke solution, as computed from rate and time.

3.2.4 Results of pre-irradiations and their application to internal quality control

A sample of the spectrophotometry record of all cells after pre-irradiation and prior to shipment is shown in table 2. The distribution of position-corrected absorbances¹² corresponding to the data in table 2 is shown in table 3 and its statistical analysis is given in table 4. Studies of this type are used to eliminate cells with very high and very low absorbances, since it cannot be ruled out that their absorbance growth with time does not show characteristics different from that of other cells.

¹¹Note that, for cobalt-60 gamma rays,

$$D_F = 1.004 D_w \approx 1.004 \times 0.869 \times \mu_{en,w} = 0.970 X$$

in air, where D_F and D_w are the absorbed doses to the Fricke solution and to water, respectively, and $\mu_{en,w}$ is the energy-absorption coefficient of water for cobalt-60 gamma rays. Thus, the effect of the quartz window of the cell amounts to a decrease in absorbed dose of roughly one percent.

¹²For details on position corrections, see section 3.1, figure 2.

Table 2. Sample of Spectrophotometry Record of All Cells After Pre-irradiation with 50 Gy of Cobalt-60 Gamma Radiation, Prior to Shipment.^a (From Data Book 837, p. 104.)

Cell No.	Position during Pre-Irrad. Row, Column	Geometry Correction Factor to Absorbance ^b	Readout Before Shipment				Average Absorbance, Geometry-Corrected ^c
			Side a		Side b		
			Absorbance	Temp. °C	Absorbance	Temp. °C	
O-1	10 , 12	1.007	0.2172	24.92	0.2173	24.92	0.2188
-2	11 , 14	1.014	0.2149	24.93	0.2129	24.90	0.2169
-3	10 , 5	1.011	0.2178	24.93	0.2166	24.94	0.2196
-4	10 , 7	1.006	0.2231	24.96	0.2219	24.93	0.2238
-5	10 , 9	1.004	0.2265	24.94	0.2226	24.93	0.2255
P-1	10 , 11	1.003	0.2231	24.94	0.2232	24.90	0.2238
-2	10 , 13	1.008	0.2188	24.92	0.2178	24.93	0.2201
-3	10 , 15	1.017	0.2189	24.95	0.2184	24.94	0.2224
-4	9 , 4	1.014	0.2205	24.94	0.2140	24.95	0.2203
-5	9 , 6	1.007	0.2188	24.94	0.2193	24.93	0.2206
Q-1	9 , 8	1.003	0.2308	24.99	0.2306	24.97	0.2314
-2	9 , 10	1.000	0.2213	24.97	0.2221	24.97	0.2217
-3	9 , 12	1.004	0.2238	24.97	0.2239	24.98	0.2248
-4	9 , 14	1.010	0.2253	24.99	0.2247	24.98	0.2273
-5	8 , 3	1.018	0.2209	25.01	0.2220	24.96	0.2254
R-1	8 , 5	1.010	0.2188	24.99	0.2189	24.97	0.2210
-2	8 , 7	1.004	0.2207	24.99	0.2243	24.97	0.2234
-3	8 , 9	1.000	0.2208	24.98	0.2203	24.98	0.2206
-4	8 , 11	1.002	0.2224	25.00	0.2231	25.00	0.2232
-5	8 , 13	1.006	0.2217	24.97	0.2211	25.00	0.2227
S-1	8 , 15	1.012	0.2163	25.03	0.2179	25.02	0.2197
-2	7 , 4	1.014	0.2159	25.03	0.2170	25.00	0.2195
-3	7 , 6	1.007	0.2232	25.04	0.2227	25.04	0.2245
-4	7 , 8	1.004	0.2188	25.02	0.2208	25.03	0.2207
-5	7 , 10	1.003	0.2192	25.01	0.2192	25.04	0.2199
etc.	etc. etc.	etc.	etc.	etc.	etc.	etc.	etc.
.
.

^aAll readings done with a slit width corresponding to 2 mm in wavelength, at a wavelength setting of 301 nm. The designations, "side a" and "side b," identify the cell windows facing the ultraviolet source.

^bThis is a correction to the exposure during pre-irradiation, which is proportional to absorbance.

^cThe small temperature fluctuations recorded did not warrant a correction.

Table. 3 Sample of Distribution of Position-Corrected Absorbances
(From table 2; see Data Book 837, p. 109.)

Absorbance Interval	Cell Designation																			
All Cells																				
0.216-0.217	M-3	X-1	0-2	D-3																
0.217-0.218	W-1	A-5	B-4	F-2	V-1	D-4														
0.218-0.219	A-3	B-1	X-5	H-5	B-5	T-3	L-3	J-4	0-1	B-3	A-4	H-3	G-2	C-2						
0.219-0.220	M-4	A-1	A-2	E-3	J-5	S-2	C-5	0-3	S-1	K-2	H-1	J-3	M-1	K-4	J-2	G-1	S-5	K-1	F-4	
0.220-0.221	G-5	P-2	K-5	C-4	L-1	D-2	F-3	P-4	M-5	U-1	V-3	R-3	P-5	H-2	L-2	S-4	N-1	N-3	N-5	E-2
0.221-0.222	T-5	H-4	R-1	W-4	W-3	W-5	U-4	U-2	F-1	Q-2	X-3	E-5	U-3	T-4	D-3	C-3				
0.222-0.223	E-1	I-3	V-4	I-1	J-1	L-5	M-2	V-2	P-3	I-4	K-3	R-5	X-2	G-3	U-5	G-4				
0.223-0.224	L-4	T-2	R-4	I-2	C-1	R-2	N-2	P-1	0-4											
0.224-0.225	F-5	S-3	D-1	Q-3	T-1															
0.225-0.226	E-4	Q-5	0-5	N-4																
0.226-0.227																				
0.227-0.228	Q-4	I-5																		
0.228-0.229	X-4																			
0.229-0.230	V-5																			
0.230-0.231																				
0.231-0.232	0-1																			
First Readout Session																				
0.216-0.217	D-5																			
0.217-0.218	A-5	B-4	F-2	D-4																
0.218-0.219	A-3	B-1	H-5	B-5	L-3	J-4	B-3	A-4	H-3	G-2	C-2									
0.219-0.220	A-1	A-2	E-3	J-5	C-5	K-2	H-1	J-3	K-4	J-2	G-1	K-1	F-4							
0.220-0.221	G-5	K-5	C-4	L-1	D-2	F-3	H-2	L-2	E-2	B-2										
0.221-0.222	H-4	F-1	E-5	D-3	C-3															
0.222-0.223	E-1	I-3	I-1	J-1	L-5	I-4	K-3	G-3	G-4											
0.223-0.224	L-4	I-2	C-1																	
0.224-0.225	F-5	D-1																		
0.225-0.226	E-4																			
0.226-0.227																				
0.227-0.228	I-5																			
Second Readout Session																				
0.216-0.217	M-3	X-1	0-2																	
0.217-0.218	W-1	V-1																		
0.218-0.219	X-5	T-3	0-1																	
0.219-0.220	M-4	S-2	0-3	M-1	S-5															
0.220-0.221	P-2	P-4	M-5	U-1	V-3	R-3	P-5	S-4	N-1	N-3	N-5	W-2								
0.221-0.222	T-5	R-1	W-4	W-3	W-5	U-4	U-2	Q-2	X-3	U-3	T-4									
0.222-0.223	V-4	M-2	V-2	P-3	R-5	X-2	U-5													
0.223-0.224	T-2	R-4	R-2	N-2	P-1	0-4														
0.224-0.225	S-3	Q-3	T-1																	
0.225-0.226	Q-5	0-5	N-4																	
0.226-0.227																				
0.227-0.228	Q-4																			
0.228-0.229	X-4																			
0.229-0.230	V-5																			
0.230-0.231																				
0.231-0.232	Q-1																			

NOTE: The cells designated Q-4, I-5, X-4, V-5, and Q-1 were eliminated.

Table 4. Statistics, from Sample Results Shown in Table 3. (See also Data Book 837, p. 108.)

Quantity	All 120 Cells	First Half (60 Cells)	Second Half (60 Cells)
Average Absorbance	0.2212	0.2205	0.2218
Standard Deviation absolute	0.0026	0.0021	0.0030
percent	1.19	0.95	1.33
Maximum Reading all cells	0.2314	0.2275	0.2314
after elimination of outliers ^a	0.2296	0.2251	0.2296
Minimum Reading all cells ^b	0.2164	0.2170	0.2164

^aSee Note on table 3

^bNone eliminated

The results shown in tables 1 and 3 are further used to obtain a check on the G-value of the particular batch of Fricke solution. The details are shown in table 5. The first quantity, the average position-corrected absorbance growth due to pre-irradiation, is calculated as the average absorbance from table 4 minus the average absorbance of the ten unirradiated cells from table 1. The third quantity, the G-value, is obtained with the aid of the first and second quantities and the experimentally determined molar linear absorption coefficient (in both SI and conventional units); it is compared with the recommended G-value [7]. The last quantity is the product of the G-value obtained for the particular Fricke solution and the linear molar absorption coefficient (see section 2.3.3) measured for our spectrophotometer, again given in both SI and conventional units; it is compared with the recommended value for this product [4]. Both the G-value obtained for our solution, our geometry, and the product ϵG are seen to be, on the average, within 0.3 percent of the respective recommended values. Therefore, the switch taken in 1986 from interpreting the absorbed dose administered by the participants by means of the measured value of ϵ , the recommended G-value, and our empirical geometry correction (see footnote 11, page 19), to interpreting the absorbed dose by means of the now recommended product, ϵG , did not affect our results significantly.

3.3 Spectrophotometry: Ensuring Long- and Short-Term Consistency and Reproducibility.

Preceding absorbance measurements, the Fricke-dosimeter cells are cleaned with distilled water, using lint-free cloths ("Optic Cloth" made by Texwipe). Currently, their absorbance is measured with a Cary, Model 219, spectrophotometer, as shown in figure 8. During the measurements ("readouts"), the temperature in the cell compartment is kept close to 25°C by means of a

relatively large-volume (0.02 m³) thermostatically controlled circulating water bath. In order to bring the temperature of the dosimeter cells close to compartment temperature prior to readout, the loaded dosimeter racks are stored for about 20 minutes in the bath compartment, above the water level. One cell at a time is then placed manually in the cell compartment for readout on both sides. The timing of the readout is computer-controlled. After a fifteen-second delay, there is a thirty-second readout period, in which fifteen absorbance readings are alternated with fifteen temperature readings. The readings are averaged automatically and stored for off-line analysis. The cell is then manually turned for readout on the other side, and the procedure is repeated, but without the initial delay. Controlling the readout schedule in this way ensures consistent absorbance readings even though complete temperature equilibrium may not have been reached.

In order to check spectrophotometer performance, each readout session is preceded by a number of spectrophotometer tests. Figure 9 and tables 6 and 7 give sample records of such tests.

Figure 9 shows the record of a determination of the wavelength corresponding to the ferric-ion absorption peak, at which the absorbances were to be read during the particular session. Shifts of a few nanometers are not unusual. (Note that the absorption peak for ferric ions given in the literature is 304 nm.)

Table 5. Check on G-Value and Product ϵG for the Particular Sample Batch of Fricke Solution (from sample data shown in table 4 and figure 10, and from eq. (3) of section 2.1).

Quantities	All 120 Cells	First Series of 60 Cells	Second Series of 60 Cells
Average position-corrected absorbance growth ΔA_{irr}	0.2212-0.0413=0.1799	0.2205-0.0413=0.1792	0.2218-0.0413=0.1805
Absorbed dose to Fricke solution (D_F)	50 Gy	50 Gy	50 Gy
G-value $\equiv 0.0977 \Delta A_{irr} / \epsilon D_F$ in units of mol J ⁻¹ ^a (using measured value of ϵ)	1.615 $\times 10^{-6}$ mol J ⁻¹ or 15.55/100 eV	1.608 $\times 10^{-6}$ mol J ⁻¹ or 15.49/100 eV	1.620 $\times 10^{-6}$ mol J ⁻¹ or 15.60/100 eV
	Value recommended by ICRU: 1.61 $\times 10^{-6}$ mol J ⁻¹ or 15.5/100 eV ^b [7]		
Product $\epsilon G \equiv 0.0977 \Delta A_{irr} / D_F$ in units of m ² kg ⁻¹ Gy ⁻¹	351.5 $\times 10^{-6}$ m ² kg ⁻¹ Gy ⁻¹	350.2 $\times 10^{-6}$ m ² kg ⁻¹ Gy ⁻¹	352.7 $\times 10^{-6}$ m ² kg ⁻¹ Gy ⁻¹
	Value recommended by ICRU: 352 $\times 10^{-6}$ m ² kg ⁻¹ Gy ⁻¹ [4]		

^aFrom ASTM Standard E 1026-84 [6].

^bFor both high-energy electrons and cobalt-60 gamma rays.

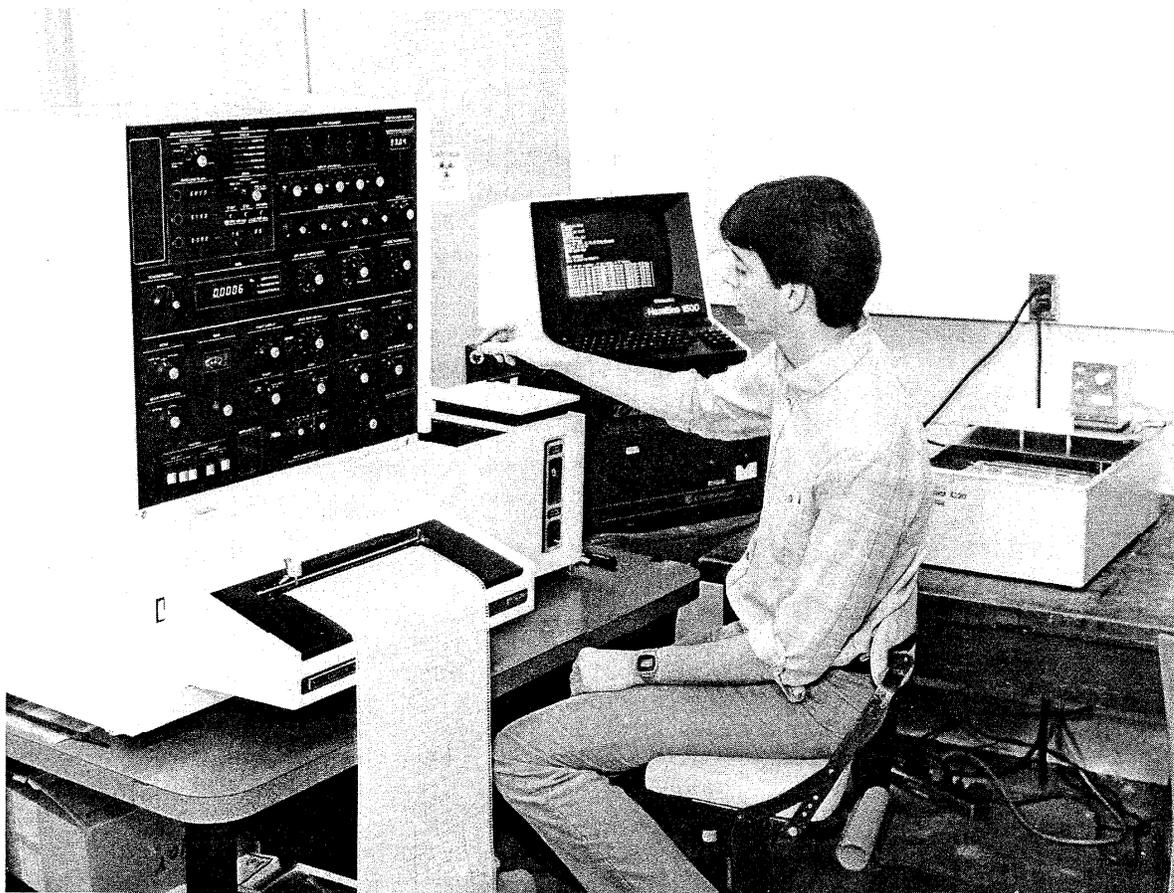


Figure 8. Spectrophotometry

Attendant is inserting spectrophotometer cell into temperature-controlled readout compartment of spectrophotometer. The circulating water bath is at right, the computer display in the center.

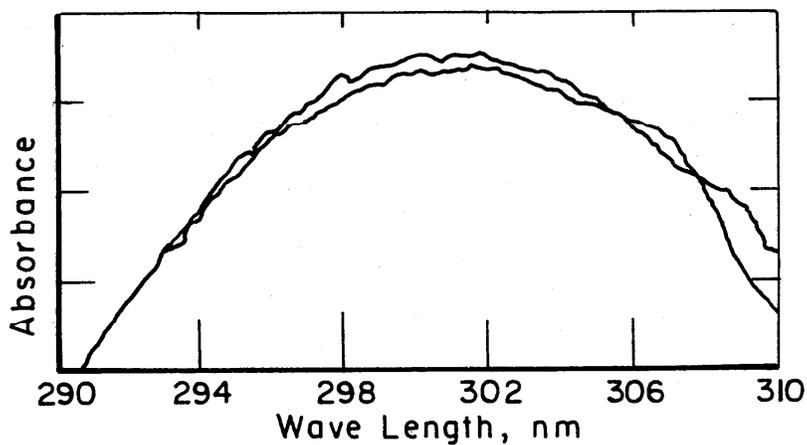


Figure 9. Sample Plot, Automatic Scan of Ferric-Ion Absorbance-vs-Wavelength Setting, Required to Establish Wavelength Setting for Absorption Peak. Spectrophotometry was done at a setting of 301 nm. (From Data Book 837, page 108.) Scan speed corresponded to 0.1 nm/s, slit width to 0.2 nm.

Table 6. Sample, Absorbances of 1 g% Potassium Nitrate Solution, Obtained as a Check on the Reproducibility of Absorbance Readings Throughout the Roughly Six Weeks Required to Complete One Test.^a (From Data Book 837, page 163.)

Readout Session	Absorbance for Cell No.				Average Absorbance	
	Z-1		Z-2			
	side a	side b	side a	side b		
Before Shipment	first session, start	.7383	.7406	.7421	.7380	.7400
	first session, end	.7389	.7413	.7421	.7384	
	second session, start	.7371	.7390	.7438	.7405	.7408
	second session, end	.7375	.7396	.7464	.7425	
After Shipment	session start	.7367	.7388	.7453	.7414	.7406
	session end	.7361	.7383	.7433	.7399	

^aAll readings done with a slit width corresponding to 2 nm in wavelength, at a wavelength setting of 301 nm. The designations, "side a" and "side b," identify the cell windows facing the ultraviolet source.

Table 7. Constancy and Linearity of Cary Spectrophotometer, Model 16, between April 1971 and April 1974. The study was carried out by measuring absorbance at two levels of concentration of potassium nitrate at the absorption peak.

Date	Peak Location nm	Net absorbance 1 g% KNO ₃	Twice net absorbance 0.5 g% KNO ₃
April 1971	302.5	0.7071 ^a	0.7073 ^a
November 1971	302.5	0.7082 ^a	0.7164
April 1972	302.0	0.7067 ^a	0.7067
November 1972	302.5	0.7060 ^a	0.7014
April 1973	302.5	0.7089 ^a	0.7094
November 1973	302.5	0.7073 ^a	0.7058
April 1974	302.0	0.7069 ^a	0.7024 ^a

^aFrom fitted graphs

Table 6 gives a sample record of the absorbances obtained on two cells filled with 1 g% potassium-nitrate solution measured before and after the two readout sessions prior to shipment of the Fricke dosimeters and before and after the readout session some six weeks later, after dosimeter return. The averages of the absorbances are seen to have remained essentially constant, indicating spectrophotometer reproducibility. Known dilutions provide a check on absorbance-scale linearity. Table 7 shows the net (background-corrected) absorbance values for a 1 g% solution and twice the corresponding values for a 0.5 g% solution, for an earlier spectrophotometer model, followed over a period of three years. Such a long-term absorbance-constancy check is facilitated if exactly the same concentration of potassium nitrate is used each time. Since the spectrophotometer in use since 1980 (Cary, Model 213) is even more reliable than the Cary, Model 16, potassium-nitrate readings later were taken only for the 1 g% solution (see table 6).

Starting in 1986, system linearity and constancy tests similar to those performed with potassium nitrate up to then, will be performed with metal-on-quartz filters, SRM 2031 [9]. Use of these filters will save time. But while they have been available since 1980, they still require further periodic checks of long-term absorbance constancy. These checks are carried out by the NBS Center for Analytic Chemistry.

3.4 Dose Interpretation and Report to Participants

The average absorbance growth for the Fricke solution contained in the spectrophotometer cells measured before and after shipment of the cells is used to compute the absorbed dose to water, D_w (in grays), administered by the participants. Using eq (4) (section 2.1), one obtains:

$$D_w = 278 (\Delta A_{irr} - \overline{\Delta A_{sp}}) \quad (5)$$

where ΔA_{irr} is the difference between the absorbance before and after shipment of the cells irradiated by the participants¹³ and $\overline{\Delta A_{sp}}$ is the average difference between the absorbance before and after shipment of the shipped and unshipped ("shipping" and "home") control cells. Sample values of $(\Delta A_{irr} - \overline{\Delta A_{sp}})$ for control cells and of individual values of ΔA_{sp} for not further irradiated cells are shown in column 6 of table 8.¹⁴ Figure 10 shows the distribution of the individual values of ΔA_{sp} from table 8, and gives the

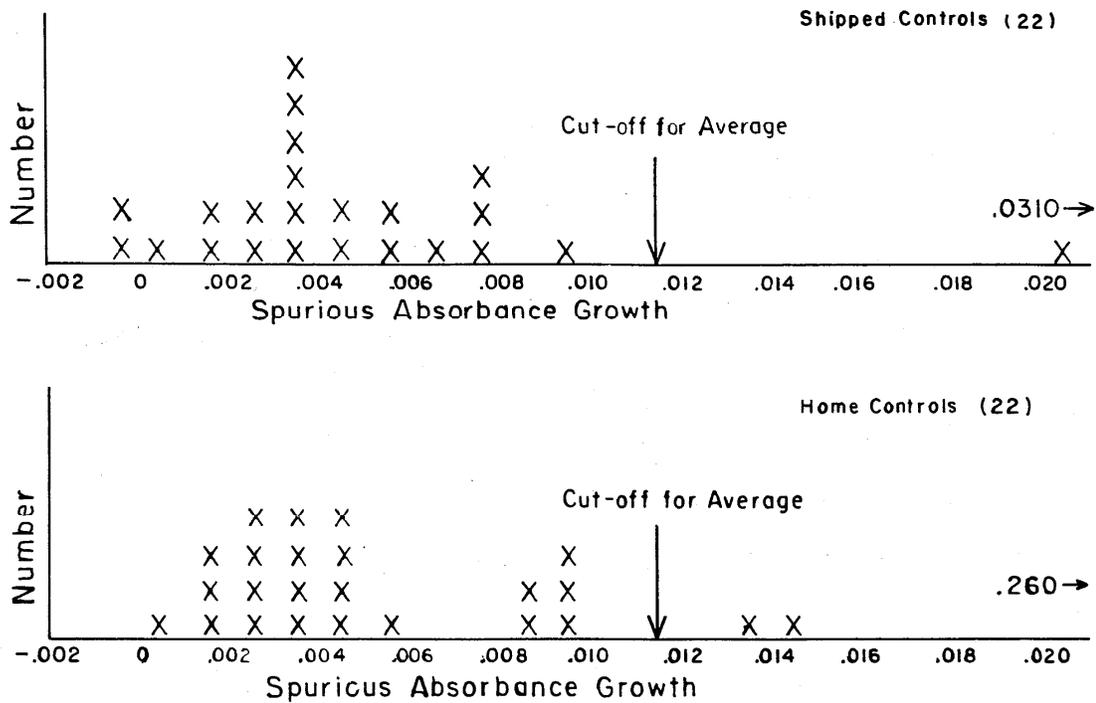
¹³Here, the absorbances before shipment are not geometry-corrected (as are those in table 2) since the absorbances of the quartz windows of the spectrophotometer cells and of the Fricke solution due to pre-irradiation drop out when the differences are formed.

¹⁴The sample record shown in table 8 pre-dates the switch to SI units for absorbed dose. It also pre-dates ICRU Report 35, recommending the use of a fixed value for the product of ϵG in the computation of absorbed dose from optical absorbance. The absorbed-dose values shown were computed with a G-value of 15.5/100 eV (non-SI) and a measured value for ϵ of 217.7 m² mol⁻¹. The complete report included in the appendix incorporates the same data.

Table 8. Sample of Spectrophotometry Record After Shipment and Associated Absorbed-Dose Interpretation.^a (From Data Book 837, p. 104 and 105.)

Cell No.	Readout After Shipment				Difference, $(\Delta A_{irr} - \Delta A_{sp})$ or ΔA_{sp}	Absorbed Dose to Water, rad		Abs. D. Difference %	Participant No.
	Side a		Side b			NBS	Particip.		
	Absorb- ance	Temp. °C	Absorb- ance	Temp. °C					
0-1	0.2216	25.13	0.2214	25.14	+ 0.0043	--	--	--	17
0-2	0.4152	25.12	0.4132	25.10	+ 0.1963	5483	5440	- 0.78	
0-3	0.2223	25.05	0.2209	25.08	+ 0.0044	--	--		18
0-4	0.4079	25.05	0.4067	24.95	+ 0.1808	5050	5000	- 0.99	
0-5	0.4081	24.89	0.4039	24.85	+ 0.1774	4956	5000	+ 0.89	
P-1	0.4275	24.86	0.4278	24.88	+ 0.2005	5600	6000	+ 7.14	19
P-2	0.4342	24.82	0.4331	24.94	+ 0.2113	5903	6000	+ 1.64	
P-3	0.4344	25.06	0.4338	25.12	+ 0.2114	5906	6000	+ 1.59	
P-4	0.4396	25.14	0.4322	25.16	+ 0.2146	5995	6000	+ 0.08	
P-5	0.2194	25.14	0.2197	25.15	+ 0.0005	--	--		
Q-1	0.2329	25.12	0.2325	25.12	+ 0.0020	--	--		
Q-2	0.2257	25.10	0.2262	25.13	+ 0.0042	--	--		
Q-3	0.2277	25.11	0.2277	25.16	+ 0.0038	--	--		20
Q-4	0.4062	25.15	0.4052	25.16	+ 0.1767	4935	4826	- 2.21	
Q-5	0.4107	25.16	0.4117	25.18	+ 0.1857	5188	4990	- 3.82	
R-1	0.4012	25.14	0.4015	25.16	+ 0.1785	4985	4840	- 2.91	
R-2	0.4074	25.17	0.4115	25.16	+ 0.1829	5110	5045	- 1.27	
R-3	0.4094	25.17	0.4088	25.19	+ 0.1845	5154	5067	- 1.69	
R-4	0.2258	25.15	0.2266	25.18	+ 0.0034	--	--		
R-5	0.2234	25.18	0.2224	25.18	+ 0.0015	--	--		21
S-1	0.2197	25.21	0.2208	25.23	+ 0.0032	--	--		
S-2	0.4366	25.19	0.4378	25.19	+ 0.2167	6054	5830	- 3.70	
S-3	0.4510	25.18	0.4505	25.24	+ 0.2238	6251	6020	- 3.70	
S-4	0.4445	25.19	0.4463	25.22	+ 0.2216	6189	6030	- 2.57	
S-5	0.4487	25.22	0.4487	25.21	+ 0.2255	6298	6120	- 2.83	

^aAll readings done with a slit width corresponding to 2 nm in wavelength, at a wavelength setting of 301 nm. The designations, "side a" and "side b," identify the cell windows facing the ultraviolet source. Note that this table was prepared prior to the switch to SI units and the use of the product ϵG for computing absorbed dose.



Obtaining the Average Spurious Growth:

	Average, ΔA_{sp}
22 shipping controls	0.0038 ± 0.0026 (stand. dev.)
22 home controls	0.0043 ± 0.0027 (stand. dev.)
all 44 controls	0.0040 ± 0.0038 (stand. dev.)
corresponding to	1.1 ± 1.1 Gy

Figure 10. Sample, Analysis of Spurious Absorbance Growth ΔA_{sp} , of Shipping and Home Controls. (From Data Book 837, p. 107.) For the assessment of the uncertainty in the correction for spurious absorbance growth (table 9, p. 42), absorbance growth was averaged over ten test series.

averages of the plotted data. The average of the growth of both shipping and home controls combined was used to compute the absorbed-dose values shown in column 7 of table 8, which — after rounding to three significant figures — is incorporated into the report sent to the participants. The report also contains plots of the distributions of the differences between assigned doses and doses reported by the participants. A sample of a complete report to the participants is included in the appendix.

3.5 Uncertainties in the Dose Interpretation

In a measurement-assurance program of the nature of the electron-dosimetry uniformity service, the uncertainties in the individual dose interpretations are of major interest. Two independent methods were used to arrive at values for the overall uncertainty. One involved a set of measurements of the differences between dose interpretations and actually delivered doses, and the other involved estimates of the uncertainties associated with the individual steps leading to a dose interpretation. The results are shown in table 9.

To arrive at the total uncertainty experimentally (first method), Fricke dosimeters were exposed at two levels of cobalt-60 gamma radiation and shipped to a number of participants who kept the packages for several days and then returned them to NBS for evaluation in the usual manner.[4] The standard deviation of the difference between the dose computed from exposure and that determined from spectrophotometry of the individual Fricke dosimeters was about 1.5 percent, corresponding to an overall uncertainty of three times this standard deviation, or ± 4.5 percent. (The "overall" uncertainty is considered to have approximately the character of a 99% confidence limit.) In the second method, the overall uncertainty in the dose interpretation was estimated from the component uncertainties involved in the individual procedural steps. In table 9, the component uncertainties not based on repeated measurements are estimated at a level of approximately one standard deviation, so that they can be treated like standard deviations. They are added in quadrature to obtain a "combined" uncertainty, which is multiplied by three to obtain the overall uncertainty. The results of the two methods are seen to be comparable.

The assessment of the uncertainties in the dose interpretation excludes the uncertainties introduced by the spectral perturbations arising from the use of quartz cells and a polystyrene phantom rather than a homogeneous water medium. This exclusion is justified in a measurement quality assurance service. If the service were ever upgraded into a calibration service, consideration would have to be given to whether or not a correction for the effect of the spectral perturbations was required, and the associated uncertainties would have to be assessed. It is expected that these uncertainties would be small compared to the uncertainty in the spurious absorbance growth between readouts, which probably could be decreased materially by the use of a sealed system. (See section 2.4.)

4. SAFETY CONSIDERATIONS

The precautions taken during the preparation, handling, and shipping of the Fricke dosimeters are those usually taken in a chemistry laboratory that handles corrosive liquids such as sulfuric acid: Old clothes and rubber

Table 9. Uncertainties in the Dose Interpretations

Procedure	Estimated Uncertainty ^a , percent
<u>First Method</u>	
Uncertainty in dose interpretation derived from repeated measurements in a specially designed experiment ^a	1.5
Overall uncertainty ^e	4.5
<u>Second Method</u>	
Uncertainty estimated for each individual step involved in the dose interpretations:	
Calibration of spectrophotometer ^b	0.2
Measurement of absorbance ^b	0.05
Correction for spurious absorbance growth between first and second readout ^c	1.5
Choice of reference value, $\epsilon G = 352 \times 10^{-6} \text{ m}^2 \text{ kg}^{-1} \text{ Gy}^{-1}$ ^b	0.6
Combined uncertainty ^(d)	1.6
Overall uncertainty ^(e)	4.8

^aAt the one standard deviation level.

^bBased on table 5.4, ICRU Report 35 [4].

^cBased on the measurement of the percent spontaneous growth for all control dosimeters in ten test series, at an average electron-dose level corresponding to an absorbance growth of 0.20 (i.e., about 56 Gy).

^dIndividual uncertainties added in quadrature.

^eThree times the uncertainty for Method 1 or three times the "combined" uncertainty for Method 2.

aprons are worn by the staff; the concentrated sulfuric acid is transported and stored in a rubber "carrying sling," and face shields are worn by the staff during pouring and weighing of the acid; acid-proof composites, glass or stainless steel, are used as supports of all containers of the Fricke solution; all liquid waste is poured into stainless-steel sinks equipped with glass-lined waste pipes; the polystyrene blocks containing the stoppered Fricke-dosimeter cells are sealed in plastic bags and packed professionally in cardboard boxes for transmittal to the participants by private carrier rather than the U.S. Mail; the box labels are marked with a note cautioning the shipper to store the boxes in the pressurized compartment of high-flying aircraft. Also, the persons handling the potassium nitrate are cautioned to handle it with care because of its potentially explosive nature.

REFERENCES*

- [1] For definitions of the radiation-dosimetry quantities and units (both SI and special) used in this document, see Radiation Quantities and Units; ICRU Report 19, International Commission of Radiation Units and Measurements, Bethesda, MD, 1971.
- [2] A Protocol for the Determination of Absorbed Dose from High-Energy Photon and Electron Beams, Task Group 21, Radiation Therapy Committee, AAPM; Med. Phys. 10(6): 741-771, 1983.
- [3] Ehrlich, M. and Lamperti, P. J. Uniformity of High-Energy Electron-Beam Calibrations, Phys. Med. Biol. 14(2), 305-314, 1969.
- [4] Radiation Dosimetry: Electron Beams with Energies Between 1 and 50 MeV; ICRU Report 35, International Commission of Radiation Units and Measurements, Bethesda, MD, 1984.
- [5] See, e.g., section 5.3.2 of ICRU Report 35 (preceding reference).
- [6] Standard Method for Using the Fricke Dosimeter to Measure Absorbed Dose in Water, ASTM Designation E 1026-84, 1985 Book of ASTM Standards, Section 12, American Society for Testing and Materials, Philadelphia, Pennsylvania, 1985.
- [7] Radiation Dosimetry: X Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 50 MeV; ICRU Report 14, International Commission on Radiation Units and Measurements, Bethesda, MD, 1969.
- [8] Mavrodineanu, R., NBS Center for Analytical Chemistry, private communication.
- [9] Mavrodineanu, R. and Baldwin, J. R., Standard Reference Materials: Metal-On Quartz Filters as a Standard Reference Material for Spectrophotometry - SRM 2031; NBS Special Publication 260-68; U.S. Government Printing Office, Washington, D.C., 1980.

Additional References to the NBS Fricke Dosimetry Service

Ehrlich, M. and Lamperti, P. J., Electron-Therapy Dosimetry, pp. 365-370, NBS Special Publication 456, Proceedings of a Symposium on the Measurements for the Safe Use of Radiation, held at NBS in March, 1976; S. P. Fivozinsky, editor; U.S. Department of Commerce, National Bureau of Standards, 1976.

Soares, C. G. and Ehrlich, M., NBS Traceability Program for Radiation Therapy, pp. 89-97, Proceedings of a Meeting on Traceability for Ionizing Radiation Measurements, held at NBS in May, 1980; H. T. Heaton II, editor; U.S. Department of Commerce, National Bureau of Standards, 1982.

*Referenced literature is available on request from the Dosimetry Group, Ionization Radiation Division, Center for Radiation Research.

Appendix 1

Forms Used in Written Exchanges with Participants

NATIONAL BUREAU OF STANDARDS

UNIFORMITY CHECK, DOSIMETRY OF HIGH-ENERGY ELECTRON-BEAMS

To All Participants:

If you plan to participate in the service, you will need a polystyrene phantom. Directions for its construction are given in the "Protocol for the Dosimetry of High Energy Electrons", SCRAD, Physics in Medicine and Biology, Vol. 11, No. 4, p. 505, October 1966, with the exception that the recess required for the dosimeter units supplied by NBS is of different dimensions from the one described in the Protocol. The enclosed figure, from NBS SP 250-4 (1986), is a picture of the unit that will be sent to you for exposure. It consists of a stoppered spectrophotometer cell filled with ferrous-sulfate solution, held firmly in place in a polystyrene parallelepiped of outer dimensions

	+0.00 cm		+0.00 cm		+0.00 cm
7.62 cm		x	5.08 cm	x	2.01 cm
	-0.03 cm		-0.03 cm		-0.03 cm

The dosimeter unit should fit snugly into the phantom recess. (It is suggested that the airspace on all sides be kept to a width of less than 0.5 mm.) The electron beam should be perpendicular to the 7.62 cm x 5.08 cm planes of the dosimeter unit. The center of the spectrophotometer cell should be in the center of the 10 cm x 10 cm field. The dosimeter may be irradiated at any phantom depth where the dose rate across the thickness of the dosimeter is known. Because the dosimeter averages absorbed dose over a depth of 1 cm, you should take this into account in your dose computation, especially below 10 MeV, since absorbed dose changes significantly over this depth, even near the depth-dose peak.

ANNOUNCEMENT TO ALL PAST PARTICIPANTS



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

<date>

Announcement of the Next Uniformity Study of Dosimetry of High-Energy Electron Beams

We plan to ship the next batch of ferrous-sulfate dosimeters between <dates>. Please let us know immediately whether or not you wish to participate, and if so, send us your purchase order before <date> and tell us how many dosimeters you require. Currently the fees are <cost> for the basic set of three dosimeters (two for irradiation and one unirradiated control), and <cost> for each additional dosimeter.

When you receive the dosimeters, please expose them according to the directions that were given to you previously. Administer between 50 and 80 Gy to water using a 10 cm x 10 cm field size. Return the dosimeters to us as soon as possible, preferably within three working days.

Please do not tamper with the dosimeter units. After exposure, place them in the zip-lock bag and seal the bag for safety reasons. Use the same packing material, or material similar to that used by us. Send the package by the same carrier as received, and mark the box, "Please ship in pressurized compartment or at low altitude". Please send return shipment pre-paid to:

Dr. Christopher Soares
C-210 Rad Phys Building
National Bureau of Standards
Route 270 & Quince Orchard Rd.
Gaithersburg, MD 20899

Sincerely,

Christopher Soares
Dosimetry Group

Note: It would be appreciated if you returned the dosimeters within three working days from the day of receipt. We are reserving the right to refuse participation in the current study if the dosimeters are not returned within ten working days after receipt.

ANNOUNCEMENT TO GROUPS THAT INDICATED THEIR INTENTION TO PARTICIPATE,
BUT DID NOT SUBMIT A PURCHASE ORDER



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

<date>

Announcement of the Next Uniformity Study of
Dosimetry of High-Energy Electron Beams

We plan to ship the next batch of ferrous-sulfate dosimeters between <dates>. Since you have already indicated your intention to participate, all that is required now is your purchase order. Currently the fees are <cost> for the basic set of three dosimeters (two for irradiation and one unirradiated control), and <cost> for each additional dosimeter.

When you receive the dosimeters, please expose them according to the directions that were given to you previously. Administer between 50 and 80 Gy to water using a 10 cm x 10 cm field size. Return the dosimeters to us as soon as possible, preferably within three working days.

Please do not tamper with the dosimeter units. After exposure, place them in the zip-lock bag and seal the bag for safety reasons. Use the same packing material, or material similar to that used by us. Send the package by the same carrier as received, and mark the box, "Please ship in pressurized compartment or at low altitude". Please send return shipment pre-paid to:

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ANNOUNCEMENT TO GROUPS THAT INDICATED THEIR INTENTION TO PARTICIPATE,
AND SUBMITTED A PURCHASE ORDER



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

<date>

Announcement of the Next Uniformity Study of
Dosimetry of High-Energy Electron Beams

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U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
GAITHERSBURG, MARYLAND 20899

REPORT OF TEST

DG <dg #/yr>

<date>

To: All Participants in the <study name> Uniformity Study of
Dosimetry in High-Energy Electron Beams

The enclosed tables and figures contain the results of the current study for all participants. A group number was assigned to each participant. The dosimeters are listed by group and not necessarily in alphabetical order. This procedure, while not giving away a participant's identity to the rest of the participants, enables each participant to check his standing among his peers with regard to accuracy as well as consistency of performance. The red arrow in the table points to the group number of the participant to whom the communication is addressed. The figures give a statistical survey of the results. Our evaluation procedure and estimated error are those discussed in "The Fricker-Dosimetry Service of the National Bureau of Standards", M. Ehrlich, NBS Special Publication 250-4. An ϵG product of $352 \times 10^{-12} \text{ m}^2 \text{ kg}^{-1} \text{ Gy}^{-1}$ was used for both electrons and cobalt-60 gamma rays. Application of the product of the formerly used values for ϵ and G would cause NBS-assigned absorbed-dose values to be larger by a factor of 1.005.

Yours sincerely,

Christopher G. Soares, Physicist
Ionizing Radiation Division
Center for Radiation Research

Enclosures

P.S. This is to remind you that our dosimeters average absorbed dose over a depth of 1 cm. At electron energies less than 10 MeV you should take this into account in your dose computation, since absorbed dose changes significantly over this depth, even near the depth-dose peak.

Group No.	Dosimeter Designation	Absorbed Dose NBS (Gy to water)	Participant (Gy)	Dose Difference (Pct. of NBS Dose)	Irradiated with:
--------------	--------------------------	---------------------------------------	---------------------	------------------------------------------	---------------------

Appendix 2. Sample of Report to Participants

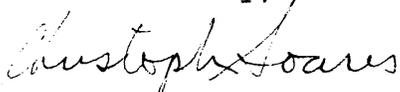
REPORT OF TEST

June 26, 1986

To: All Participants in the Spring 1986 Uniformity Study of
Dosimetry in High-Energy Electron Beams

The enclosed tables and figures contain the results of the current study for all participants. A group number was assigned to each participant. The dosimeters are listed by group and not necessarily in alphabetical order. This procedure, while not giving away a participant's identity to the rest of the participants, enables each participant to check his standing among his peers with regard to accuracy as well as consistency of performance. The red arrow in the table points to the group number of the participant to whom the communication is addressed. The figures give a statistical survey of the results. Our evaluation procedure and estimated error are those discussed in "The Fricke-Dosimetry Service of the National Bureau of Standards", M. Ehrlich, NBS Special Publication 250-4 (1986). An ϵ G product of $352 \times 10^{-6} \text{ m}^2 \text{ kg}^{-1} \text{ Gy}^{-1}$ was used for both electrons and cobalt-60 gamma rays. Application of the product of the formerly used values for ϵ and G would cause NBS-assigned absorbed-dose values to be larger by a factor of 1.005.

Yours sincerely,



Christopher G. Soares, Physicist
Ionizing Radiation Division
Center for Radiation Research

Enclosures

P.S. This is to remind you that our dosimeters average absorbed dose over a depth of 1 cm. At electron energies less than 10 MeV you should take this into account in your dose computation, since absorbed dose changes significantly over this depth, even near the depth-dose peak.

Report of Test, Continued

Group No.	Dosimeter Designation	Absorbed Dose		Dose Difference (Pct. of NBS Dose)	Irradiated with:
		NBS (Gy in water)	Participant (Gy)		
1	A-2	55.2	56.42	+2.3	8 MeV el
	A-3	62.0	63.27	+2.2	13 MeV el
	A-4	59.1	60.78	+2.9	10 MeV el
2	B-1	49.4	51.00	+3.4	30 MeV el
	B-2	49.4	50.60	+2.6	7.5 MeV el
	B-3	48.6	50.00	+3.0	6 MeV el
	B-5	49.2	50.00	+1.7	12 MeV el
	C-1	50.7	50.00	-1.3	22 MeV el
3	C-3	59.1	50.40	-14.7	12 MeV el
	C-4	51.9	50.50	-2.5	20 MeV el
4	D-2	50.0	49.95	+0.1	6 MeV el
	D-3	52.1	49.95	-4.1	12 MeV el
	D-4	53.0	49.95	-5.6	15 MeV el
5	E-1	40.6	47.95	+18.1	6 MeV el
	E-2	45.4	47.95	+5.6	9 MeV el
	E-3	66.9	49.95	-25.3	13 MeV el
	E-4	50.3	49.95	-0.6	17 MeV el
	F-1	51.4	49.95	-2.6	20 MeV el
6	F-2	60.7	60.00	-1.1	10 MV xrv
	F-3	59.9	60.00	+0.3	6 MeV el
	F-4	61.2	60.00	-1.9	9 MeV el
	G-1	59.9	60.00	+0.3	12 MeV el
	G-3	59.7	60.00	+0.5	15 MeV el
	G-4	60.6	60.00	-0.9	18 MeV el
	G-5	60.4	60.00	-0.6	Cobalt-60
7	H-3	52.7	54.20	+3.0	7 MeV el
	H-4	52.4	54.92	+4.8	18 MeV el
	H-5	57.5	54.05	-5.9	12 MeV el
8	I-2	52.4	50.00	-4.4	9 MeV el
	I-3	50.5	50.00	-0.9	12 MeV el
9	J-1	60.1	60.00	-0.1	6 MeV el
	J-2	59.8	60.00	+0.5	15 MeV el
10	J-4	57.0	57.87	+1.7	Cobalt-60
	J-5	55.0	56.89	+3.5	Cobalt-60
11	K-2	49.0	49.00	+0.2	8 MeV el
	K-3	52.6	49.04	-6.7	10 MeV el

Report of Test, Continued

Group No.	Dosimeter Designation	Absorbed Dose		Dose Difference (Pct. of NBS Dose)	Irradiated with:
		NBS (Gv in water)	Participant (Gv)		
12	L-1	59.4	60.00	+1.1	10 MeV el
	L-2	60.9	60.00	-1.3	10 MeV el
13	L-4	56.5	50.00	-11.5	7 MeV el
	L-5	53.5	50.00	-6.5	10 MeV el
	M-1	50.6	50.00	-1.0	12 MeV el
14	M-3	60.5	63.68	+5.3	9 MeV el
	M-4	58.3	57.90	-0.5	12 MeV el
	M-5	66.0	61.96	-6.0	18 MeV el
15	N-2	51.3	50.00	-2.4	7 MeV el
	N-3	50.8	50.00	-1.4	10 MeV el
	N-4	51.1	50.00	-2.0	15 MeV el
	N-5	50.8	50.00	-1.5	20 MeV el
16	O-1	66.5	63.49	-4.5	10 MV xrv
	O-3	56.3	52.26	-7.1	9 MeV el
	O-4	62.6	63.52	+1.6	12 MeV el
	O-5	64.7	65.57	+1.4	15 MeV el
	P-2	34.3	49.64	+44.9	6 MeV el
17	P-4	61.1	59.85	-2.0	7 MeV el
	P-5	63.8	63.00	-1.2	10 MeV el
	Q-1	61.8	63.00	+2.0	12 MeV el
18	Q-3	51.2	50.00	-2.3	15 MeV el
	Q-4	49.1	50.00	+1.9	20 MV xrv
	Q-5	51.4	50.00	-2.6	6 MV xrv
19	R-2	60.2	60.70	+0.9	10 MV xrv
	R-3	48.4	50.00	+3.5	12 MeV el
	R-4	50.5	50.00	-0.9	6 MeV el
20	S-3	56.8	50.00	-11.9	17 MeV el
	S-4	49.6	50.00	+1.0	9 MeV el
21	T-4	51.4	50.00	-2.7	12 MeV el
	U-1	51.4	50.00	-2.6	14 MeV el
22	U-2	56.6	55.97	-1.0	12 MeV el
	U-4	56.0	57.29	+2.5	7 MeV el
23	V-2	49.4	50.00	+1.4	10 MeV el
	V-3	50.4	50.00	-0.8	14 MeV el
24	V-4	49.9	50.00	+0.3	9 MeV el
	V-5	56.1	50.00	-10.9	20 MeV el

